

**EFFECT OF CONCENTRATION BY SERUM-PULP  
METHOD ON VALENCIA ORANGE JUICE  
PROPERTIES AND ITS VOLATILE FLAVOR  
COMPOUNDS**

**Rabie M.A.; Bassiuny S .S.; Siliha H.I. and Abou El-Maaty S.M.  
Department of Food Science, Faculty of Agriculture,  
Zagazig University, Zagazig, Egypt.**

*Received 5 / 4 / 2003*

*Accepted 20 / 4 / 2003*

**ABSTRACT:** Orange juice concentrate is one of the most important orange products, which is widely appreciated in the world market. Concentration of orange juice by evaporation alters the final color, taste, aroma and nutritional characteristics of reconstituted juice. The use of enzymatic treatment in combination with serum pulp method for concentration of Valencia orange juice and the impact of this process on chemical composition and volatile flavor compounds were investigated. The physicochemical properties of orange juice, pulp and serum varied widely. The ratio of glucose, fructose and sucrose was found to be 1:1:2. Pulp particles were rich in vitamin C and were characterized by the highest color values. Quantitative determination of volatile flavor compounds of Valencia orange juice showed that, in total, there were 24 compounds of which 9 compounds were present in concentrations higher than 0.1 ppm such as acetaldehyde, ethanol,  $\alpha$ -pinene, ethylbutanoate, limonene, linalool, citronellal, valencene and  $\alpha$ -terpineol. Ten compounds were present in concentrations lower than 0.1 ppm, these were methanol, ethylacetate, iso-ethylbutanoate, hexanal, ethylhexanoate, trans-2-hexenal, 1-hexenal, pelargonaldehyde, geranial and neral. Additionally, there were 5 unknown compounds. Distribution of volatile flavor compounds between pulp and serum showed that,  $\alpha$ -pinene, limonene, valencene, linalool and citronellal are contained mainly in the pulp particles, while acetaldehyde, ethanol and ethylbutanoate were present mainly in the serum. Comparison of

**volatile flavor compounds of unpasteurized juice, reconstituted juice from concentrate produced by conventional evaporation process and reconstituted juice from concentrate produced by serum-pulp method indicated that, fresh-unpasteurized juice contained the highest concentration of volatile flavor. Reconstituted juice from serum-pulp method had higher concentration of volatile flavor than that from conventional evaporation. Data presented in this study provide evidence that concentration by serum-pulp method is a promising process for production of high quality juice.**

**Keywords:** Orange juice, Orange juice concentrate, Chemical composition, Enzymatic treatment, Serum-pulp method, Volatile flavor compounds.

## **INTRODUCTION**

Orange juice is considered as an excellent source of vitamin C and is a desired product by many consumers who are interested in maintaining healthy diet. It has been produced in several forms such as fresh-squeezed unpasteurized juice, orange juice from concentrate (reconstituted juice), pasteurized juice and frozen concentrate. Consumers nowadays prefer orange juice which is as natural as possible and without extensive processing. Although, market research in Europe indicated that the use of fresh-squeezed, unpasteurized orange juice has increased by 50 % a year (Decio, 1993). The use of orange juice concentrate is still holding the principal share in the world

market. Orange juice produced from concentrate has been subject of numerous studies (Fellers et al., 1986; Marcy et al., 1989 and Rassis and Saguy, 1995) and it is obvious that, concentration process alters the final taste, aroma and nutritional characteristics. Asker and Treptow (2000) reported that, most aroma components and heat sensitive vitamins are lost during evaporation in addition to generation of off flavors. There are alternative concentration processes other than the use of evaporation technique such as freeze concentration and reverse osmosis, but they have serious drawbacks. Freeze concentration process causes juice loss due to the inclusion of juice solids with the ice mass. Reverse osmosis encounters problems of severe

concentration polarization and loss of aroma compounds. Moreover, both freeze concentration and reverse-osmosis have relatively high process costing (Pala and Bielig, 1978 and Deshpande et al., 1984). Concentration by serum-pulp method may provide a better way for preserving quality characteristics of orange juice. It is carried out by centrifugal separation of the juice into serum and pulp. The serum is concentrated by evaporation to high degree of soluble solids and recombined with the pulp to produce orange juice concentrate. The main idea of serum-pulp method is based on the assumption that, most aroma and heat sensitive compounds in turbid juices are localized in the pulp and that the low viscosity of serum facilitates concentration to high soluble solids and reduce flavor and nutrient deteriorations and browning reactions. It was first described by Pimazzoni (1961) for the production of tomato concentrate and then used for the concentration of other fruit juice concentrate such as orange juice (Peleg and Mannheim, 1970 and Johnson, 1993) and mango concentrates (Abd El-Hadi, 2002). It has long been believed that, the delicate and

desirable flavor of fresh-squeezed orange juice is due to a complex mixture of many volatile constituents blended in proper proportions (Shaw et al., 1994). The number of these constituents in orange juice samples varies widely in the literature depending on orange variety, type of juice (fresh or reconstituted), method of juice extraction, method of volatile extraction (distillation or headspace) and the method and the conditions employed for flavor analysis. Moshonas and Shaw (1992) compared static and dynamic headspace technique and quantified 16 constituents and Moshonas and Shaw (1994) quantified 46 volatile constituents of both hand extracted and mechanically extracted fresh orange juice samples from 5 different cultivars. In other limited studies, Pino (1982) quantified seven volatile constituents and Rodrigues and Culberston (1983) quantified eight volatile constituents each involving a single sample of fresh orange juice.

The objectives of the present study was to evaluate the use of enzymatic treatment together with serum-pulp method for concentration of Valencia orange juice and to investigate the

impact of this process on the Physicochemical composition and volatile flavor constituents of the reconstituted orange juice.

## MATERIALS AND METHODS

**Materials:** Orange fruits *Citrus sinensis* var. Valencia were harvested from a private farm in Banattf, Zagazig, at the maturity stage. Orange fruits were harvested in the early morning and transported to the laboratory of Food Science Department, Faculty of Agriculture, Zagazig University. The fruits were washed with tap water and cut into two halves.

### **Orange Juice Processing:**

The juice was extracted using a laboratory juice extractor operated by hand-reaming. The expressed juice was passed through a single layer of muslin cloth to filter out the seeds, solids and pulp materials.

### **Separation of Orange Juice into Pulp and Serum:**

Serum of fresh orange juice was separated by centrifuging fresh orange juice at 4500 rpm for 20 min using a Sigma Labor Centrifuge (D-37520 modell 2-15, Germany).

### **Concentration of Orange Juice and Serum:**

Unpasteurized orange juice as well as serum was concentrated by a vacuum rotary evaporator under vacuum (Buechi, B-16 g Vacuum System Switzerland). Concentration was carried out at 60°C on untreated serum and serum previously treated with 20, 40 and 60 ppm of pectintranseliminase (Novo nordisk, Ferment Dittingen, Switzerland. Enzyme reaction was performed at 25°C for 30 min and followed by enzyme inactivation at 90°C for 5 min. After concentration, the concentrated serum was recombined with the pulp to give serum pulp concentrate. Samples of fresh squeezed orange juice was pasteurized and concentrated by evaporation to give orange juice concentrate by the conventional evaporation method.

Reconstitution of orange juice concentrate into single strength juice was carried out using condensate resulted during concentration.

### **Physico-Chemical Analysis of Orange Juice:**

Total soluble solids, pH value, titratable acidity and free amino nitrogen of orange juice were determined as described by

AOAC (1984). The color index values were measured at the Laboratory of Food Technology, National Research Center, Dokki, Cairo, using a Hunter Lab (modell) D25 color difference meter according to Askar and Treptow (1993). Cloud stability of fresh orange juice was measured in the separated serum at 660 nm as described by Askar and Treptow (1993). Viscosity of orange juice and serum was measured as described by Askar and Treptow (1993). The enzyme kit (Boehringer and Mannheim) was used to determine the concentration of D-glucose and D-fructose. Sucrose in orange juice was determined as described by Matissek et al., (1992). Ascorbic acid was determined by the method of Henniger (1981) in orange juice samples.

#### ***Analysis of Volatile Flavor Compounds:***

The determination of volatile flavor compounds were carried out by Headspace Gas chromatographic Technique. Five grams of orange juice or concentrated serum was weight in 20 ml headspace-vail and kept in a shaker for 5 min, and finally tempered at 80 °C for 40 min. 250 microliter headspace was injected

in the capillary gaschromatograph HP5890, serie II with split-Splittless injector which was equiped with capillary column (supelco) 30 m x 0.25 mm. Temperature of the flame-ionization detector was 250°C and injection port was 250°C. Oven temperature program was programmed as follows: 50°C hold for 2 min, from 50 to 70 °C at 10°C /min, at 70°C hold for 5 min and from 70°C to 135°C at 2°C /min. Helium was used as carrier gas at flow rate of 1ml/min. Quantitative deter-mination was carried out using external standard of the key volatile flavor compounds of orange juice.

## **RESULTS AND DISCUSSION**

**Physicochemical Properties of Orange juice, Pulp and Serum:** From the physical point of view, orange juice is a suspension of fine pulp particles suspended in a clear aqueous phase (serum). The ratio of serum and pulp obtained by centrifugal separation of unpasteurized Valencia orange juice was found to be 9:1.

Table 1 shows total soluble solids (TSS), sugars, titratable acidity pH, free amino nitrogen

and vitamin C contents as well as the color values L, a and b of unpasteurized Valencia orange juice, pulp and serum separated from it by centrifugation. Total soluble solids (TSS) of unpasteurized Valencia orange juice and its serum was found to be 11.20 and 10.0°Brix, respectively. Farnworth et al., (2001) reported similar TSS content (11.60°Brix) in unpasteurized Valencia orange juice from Mexico. It is well known that, most of the TSS in orange juice are sugars, mainly glucose, fructose and sucrose. Glucose and fructose contents of orange juice was similar, being 2.45 and 2.40%, respectively indicating that, the ratio of glucose to fructose is almost 1:1. Sucrose constituted the highest sugar concentration, being 4.23%. These results are in harmony with those reported by several authors, while Moukarzel and Sabri (1996) found that. these values were, respectively, 2.40, 2.40 and 4.70%. Farnworth et al., (2001) reported that, glucose, fructose and sucrose concentration of unpasteurized Valencia orange juice was 1.93, 2.32 and 4.90 g/100 ml. More recently, Brereton (2002) reported that, the principal sugars glucose, fructose and sucrose in orange juice are present in the ratio 1:1:2. As expected, pulp particles contained lower concentration of soluble sugars than the serum. The concentration of glucose, fructose and sucrose of pulp and serum were 1.36 and 2.25, 1.99 and 2.20 and 4.01 and 4.01%, respectively. It should be noticed that, after centrifugal separation of the juice into pulp and serum, the pulp was not washed to release the residual serum adhering to the pulp particles. Therefore, most of the soluble sugars of the pulp are originally belonging to the serum. Titratable acidity of unpasteurized Valencia orange juice and its serum reached 1.11%. The pH value of unpasteurized Valencia orange juice and serum was found to be 3.33. Free amino nitrogen content, which is a measure of free amino acids and other nitrogenous compounds was found to be 32.25 mg/100 ml in the unpasteurized Valencia orange juice. Vitamin C content of orange juice, pulp and serum accounted for 55.61, 42.38 and 13.23 mg / 100 gm, indicating that pulp particles are rich in vitamin C content. The color values determined by Hunter system is presented in Table 1. The pulp particles were characterized

by the highest L lightness (61.39), a (redness to greenness, 17.35) and b (yellowness to blueness, 75.62). Unpasteurized orange juice, on the other hand, had lower values for L (52.32), a (8.99) and b (52.72). These results are confirmed by the fact that, juice color is a result of reflection of light by the pulp particles (Gennovese et al., 1977) and changes in suspended pulp particles or its content in the juice cause changes in the color determined by the color difference meter. In line with that, serum which is free from pulp particles was not subjected to color measurement by this method.

#### **Volatile Flavor Constituents of Unpasteurized Orange Juice, Pulp and Serum:**

The delicate and desirable flavor of fresh squeezed orange juice is due to a complex mixture of volatile constituents blended naturally in the proper proportions. Some of these are water soluble originating from the juice in the juice sacs while others are oil-soluble originating from juice oil and peel oil. The volatile compounds in the juice are distributed between serum and pulp particles. Table 2 lists the quantitative values of volatile flavor constituents of

unpasteurized Valencia orange juice, pulp and serum separated from it by centrifugation. Twenty four compounds were quantitatively identified. Some of these compounds which are accurately identified were present in concentration higher than 0.1 ppm such as acetaldehyde, ethanol,  $\alpha$ -pinene, ethylbutanoate, limonene, citronellal, linalool, valencene and  $\alpha$ -terpineol. There were five compounds which were quantified but not accurately identified and regarded as unknown. The remaining compounds (10 compounds) were present at concentrations lower than 0.1 ppm which was the lowest limit for quantification employed in this study. These latter compounds are well known to contribute to the volatile flavor of orange juice. Acetaldehyde was present in the juice at concentration of 6.3 ppm while in pulp and serum 5.6 and 5.7 ppm were found, respectively. This indicates that, most of acetaldehyde are contributed by the serum. The concentration of acetaldehyde is in accordance with the value reported by Velez et al., (1993) who found that, the concentration of acetaldehyde ranged between 3-7 ppm in fresh orange juices.

Acetaldehyde was identified in the aroma profile of grapefruit, orange, lime, lemon and calamondin (Nisperos-Carriedo et al., 1992; El-Samahy et al., 1999 and Farnworth et al., 2001). Shaw (1991) considered acetaldehyde as a major contributor to fresh orange flavor. Ethanol constituted the highest concentration of the volatile constituents in unpasteurized Valencia orange juice (411 ppm) and serum (522 ppm). In pulp, ethanol comes as the second largest compound with a concentration of 422 ppm. Similarly, Nisperos-Carriedo and Shaw (1990a) and Farnworth et al., (2001) found that, ethanol was the highest alcohol concentration in fresh orange juice, accounting, respectively, 420 and 615 ppm. According to Shaw (1991), ethanol may act as a solvent that gives lift to other aroma, but does not contribute any aroma of its own. Its aroma threshold range between 100 ppm (Fazzalari, 1978) and 1150 ppm (Pino et al., 1986) confirming its minor contribution to aroma of fresh orange juice. The concentration of  $\alpha$ -pinene in unpasteurized orange juice and pulp was found to be 0.4 and 2.1 ppm, respectively, while it was found in the serum as traces. This

indicates that,  $\alpha$ -pinene is contributed mainly by the pulp particles. Its concentration in fresh orange juice varies widely in literature, being 0.10 ppm (Nisperos-Carriedo and Shaw, 1990b), 0.10 to 1.09 ppm (Moshonas and Shaw, 1994) and 5.44 ppm Farnworth et al., (2001). It is a constituent of peel oil and of juice oil, therefore its level depend on the oil content in the juice. The odor of  $\alpha$ -pinene was described as green, floral and lemon like flavor (Shaw, 1979) and was judged by Ahmed et al., (1978) to make positive contribution to flavor of processed orange juice. Its aroma threshold ranged between 9.5 ppb (Ahmed et al., 1978) and 62 ppb (Pino et al., 1986). Ethylbutanoate was quantified only in the unpasteurized juice, being 0.1 ppm, while it was present in trace amounts in pulp and serum (Table 2). It is present in most juices in amounts above the aroma threshold values (0.13 ppb) and (1.1 ppb) reported by Ahmed et al., (1978) and Pino et al., (1986) respectively. According to Moshonas and Shaw (1986) ethylbutanoate is one of the most important contributor to desirable orange flavor and present at 7 times its flavor threshold in Valencia orange juice.



Limonene represented the second most abundant volatile component in unpasteurized Valencia orange juice, being 119 ppm (Table 2). In the pulp it made up the highest volatile component (734 ppm) while, only trace amount was detected in the serum, indicating that pulp is the prime contributor of limonene. Limonene exhibits distinctive aroma of lime (Lindsay, 1985) and is considered the most abundant aroma component in all citrus fruits. Its aroma threshold was found to be as low as 10 ppb (Belitz and Grosch, 1987). The optimum level of limonene in processed orange juice has been estimated from the optimum total oil level to be between 150-220 ppm (Carter, 1990). The concentration of linalool in unpasteurized Valencia orange juice accounted for 3.1 ppm. Pulp portion contained higher concentration (5.2 ppm) than that found in the serum (2.5 ppm), indicating that pulp is a major contributor of linalool. Our result is in agreement with data reported by (Moshonas and Shaw (1994) who found that, the concentration of linalool ranged between 0.013 and 3.7 ppm in several varieties of oranges (Valencia, Pineapple, Hamlin, Navel, Pera and

Ambersweet). On the other hand, Farnworth et al.,(2001) obtained much higher concentrations (8.45 ppm). Linalool was found to be the most important compound in fresh lemon oil (Schieberle and Grosch, 1988) and important contributor to orange juice flavor (Moshonas and Shaw, 1994). Its low threshold value (4.7 ppb; Pino et al., (1986) suggests that, it is an important contributor in orange juice. Similarly valencene was found to be present mainly in the pulp particles, being 5.2 ppm (Table 2). Its concentration in the unpasteurized juice was 0.4 ppm while only trace amount was detected in the serum. In their analysis of volatile flavors in 72 orange juices from most widespread blond and blood cultivars, Maccarone et al., (1998) found that, valencene contribute powerful citrusy top notes. Nisperos-Carriedo and Shaw (1990a) quantified valencene in fresh orange juice to be 8.0 ppm, while Moshonas and Shaw (1994) found an average of 0.83-12.1 ppm. The concentration of  $\alpha$ -terpineol in unpasteurized orange juice, pulp and serum was found to be 1.1, 1.3 and 0.4 ppm, respectively. In heat treated citrus juice,  $\alpha$ -terpineol is formed from

the degradation of limonene and linalool. It is described as being musty, earthy and mushroom-like note in deteriorated lemon flavor (Pfannhauser, 1989) and yet, pure  $\alpha$ -terpineol is described as delicately floral and lilac (Arctander, 1969). Citronellal was detected in pulp portion at a concentration of 0.4 ppm, while in the juice and serum traces were identified. Data in Table 2 also show that, methanol, ethylacetate, iso-ethylbutanoate, hexanal, ethylhexanoate, trans-hexenal, 1-hexanal, pelargonaldehyde, geranial and neral which are important contributors to fresh orange juice aroma are detected in concentration less than 0.1 ppm, the minimum limit for volatile quantification used in this study. Moreover, four of the five unknown compounds were found to be present mainly in the pulp portions, suggesting their importance as flavor contributors of fresh orange juice. Based on the above mentioned flavor profile of orange juice, pulp and serum, it is to be concluded that  $\alpha$ -pinene, limonene, valencene, linalool, citronellal are contained in the pulp portion, while acetaldehyde, ethanol and ethylbutanoate are mainly contributed by the serum.

### **Concentration by Serum - Pulp Method:**

During concentration and due to the presence of hydrocolloid (pectin) in the serum, the viscosity increases and concentration to high degree total soluble solids is altered. Therefore, enzymatic treatment to depectinize the serum was performed by using pectintranseliminase (PTE). This enzyme causes trans-eliminative cleavage of the 1-4  $\alpha$ -glycosidic linkage in pectins, resulting in the formation of double bond between C4 and C5 at the non reducing end of one of the reaction product. It attacks preferably at random, highly esterified pectin which results in rapid reduction in viscosity. The effect of different enzyme concentrations on some parameters of treated serum is compared to untreated presented in Table 3. Total soluble solids and titratable acidity remained unchanged. Serum viscosity was not affected at enzyme concentration of 20 ppm, while it dropped to 5.60 and 4.90 centi-poise at enzyme concentration of 40 and 60 ppm, respectively. The maximum soluble solids obtained from the concentration of untreated serum was 72°Brix, while the use of enzymatic

treatment at 40 ppm pectintranseliminase gave the highest TSS in serum concentrate (80°Brix). Johnson (1993) obtained similar TSS in Valencia orange juice.

Table 4 shows the changes in volatile flavor constituents of concentrated serum prepared by serum - pulp method. It can be seen, that the concentration of acetaldehyde was decreased from 16.0 ppm in untreated serum to 4.8 ppm in enzymatically treated serum. Ethanol was decreased from 15.0 ppm to 2.7 ppm. Similarly, the concentration of limonene found in enzymatically treated serum was half the concentration observed in untreated serum concentrate. On the other hand, valencene concentration was found to be 2.0 ppm in enzymatically treated serum, while it was present as trace amount in untreated one. It is worthwhile noting that, the changes in volatiles flavor compounds of the serum concentrate most probably due to the heat treatment (90°C for 5 min) used to inactivate the enzyme. Untreated serum concentrate was prepared from unpasteurized juice. Comparing the results presented in Table 2 and Table 4, it is clear that

concentration process by evaporation dramatically affected the concentration of the very volatile compounds such as acetaldehyde, ethanol and ethyl butanoate, while the less volatile compounds such as limonene and valencene were not affected. These results are confirmed by data reported in the literature for the concentration of citrus juices (Askar et al., 1981 and El-Samahy et al., 1999).

Data presented in Table 5 shows the volatile flavor compounds of reconstituted orange juice prepared by conventional evaporation and by serum-pulp method and compared to unpasteurized orange juice. The final concentration of orange juice concentrate resulted from the conventional method was 72°Brix, while that prepared by serum-pulp method had final concentration of 80°Brix. Unpasteurized orange juice contained the highest concentration of volatile flavor compounds as compared to reconstituted orange juice prepared from concentrates and from the pasteurized juice. This indicates the adverse effect of concentration process on the characteristic volatile flavor compounds of orange juice. On the other hand,

reconstituted orange juice prepared by conventional method of concentration had the lowest concentration of volatile flavors, indicating the advantage of using serum-pulp method for concentration of orange juice. The slight differences in concentration of volatile flavor compounds between untreated and enzymatically treated concentrate is ascribed to heat treatment for enzyme inactivation. It is to be concluded that, the use of serum-pulp method in combination with enzymatic treatment is a promising technique since it gives concentrates having higher soluble solids content and better aroma profile than that obtained from conventional method of concentration.

### REFERENCES

- A.O.A.C. (1984). Official Methods of Analysis. 13<sup>th</sup> edition, Association of Official Analytical Chemists, Washington, D. C.
- Abd El-Hadi, M.M. (2002). Technological studies on some fruit products. Ph. D.Thesis, Faculty of Agric., Zagazig Univ.Egypt. *Acta Alimentaria*, 11 (1): 1-9.
- Ahmed, E.M.; Dennison, R.A.; Dougherty, R.H. and Shaw, P.E. (1978). Flavor and odour thresholds in water of selected orange juice components. *J. Agric. Food Chem.*, 26, 187-191.
- Arctander, S. (1969). *Perfums and flavor chemicals. Allured: Carol Stream, Vol. I and II.*
- Askar, A.; El-Samahy, S.K.; Abd El-Baki, M.M; Ibrahim, S.S and Abd El-Fadeel, M.G. (1981). Production of lime juice concentrates using serum-pulp method. *Alimenta*, 20, 121-128.
- Askar, A. and Treptow, H. (1993). Quality assurance in tropical fruit processing. Springer Verlag Berlin Heidelberg, Germany. pp 57,60.
- Askar, A. and Treptow, H. (2000). Critical points on mango processing. In proceeding of the 23<sup>rd</sup> Symposium of International Federation of fruit juice producers (IFU) pp.53-81
- Belitz, H.D. and Grosch, W. (1987). In *Food Chemistry*, Springer Verlag.
- Brereton, P. (2002). Fruit juice authenticity. Department for Food & Rural Affairs (DEFRA).
- Carter, R.D. (1990). Florida citrus juice from concentrate Production, packaging and distribution; Technical Manual, Florida Department of Citrus, Lcke, Alfred, FL.

- Decio, P. (1993). The new trend towards freshly squeezed orange juice. *Fruit Processing*, 3, 238-239.
- Deshpande, S.; Sathe, K. and Salunke, K. (1984). Freeze concentration of fruit juice. *CRC Critical Rev. Food Sci.Nutr.*, 20, 173-248.
- El-Samahy, S.K.; Hayam, Allam;Ghonheim,S.I.;Askar, A and Siliha, H. (1999). Effect of pasteurization and concentration on volatiles of lime juice. *Proc. Of the 22<sup>nd</sup> IFU symposium, Paris 15-19 March, Page 44-56.*
- Farnworth, E.R; Lagacé, M.; Couture, R.; Yaylayan, V. and Stewart, B. (2001). Thermal processing, storage conditions and the composition and physical properties of orange juice. *Food Research International*, Vol. 34 (1) : 25-30.
- Fazzalari, F.A. (1978). *Compilation of odor and taste thresholds data ASTM Data Series DS48A; American Society for Testing and Materials. Philadelphia.*
- Fellers, P. J.; De Jager, G. and Poole, M. J.(1986). Quality of Retail Florida-Packed Frozen Concentrated Orange Juice as Determined by Consumers and Physical and Chemical Analyses. *Journal of Food Science*, 51 (5): 1187-1190.
- Gennovese, D.B.; Elustodo, M.P. and Lozano, J.E. (1977). Color and cloud stabilization in clouding apple juice by stream heating during crushing. *J. Food Sci.*, 62:1171-1175.
- Henniger, G. (1981). *Enzymatische Bestimmung von L Ascorbinsaeure in Lebensmitteln, Pharmazeutika und biologischen Fluessigkeiten, Alimenta*, 20, 12-14.
- Johnson, J.R. (1993). Technical and economical feasibility of a non conventional methods for concentrating orange juice. *Ph.D Thesis, Florida University.*
- Lindsay, R.C. (1985). *Flavors. In Food chemistry, Fennema O.R. Marcel Dekker, pp 585-628.*
- Maccarone, E.; Campisi, S.; Fallico, B ; Rapisarda, P. and Sgarlata, R. (1998). Flavor components of Italian orange juices. *J. Agric. Food Chem.*, 46 (6): 2293-2298.
- Mannheim, C. H. and Passy, N. (1977). Recovery and concentration of citrus aroma. *International Citrus Congress. Orlando, Florida, International Society of Citriculture. 3: 756-762..*

- Marcy, J.E; Hansen, A.P and Graumlich, T.R. (1989). Effect of storage temperature on the stability of aseptically packaged concentrated orange juice and concentrated orange drink. *J. Food Sci.*, 54, :227-230.
- Matissek, R.; Schnepel, F.M. and Steiner, G. (1992). *Lebensmittel-analytik Grundzuege. Methoden. Anwendungen. 2.Auflage.* Springer Verlag, Berlin/Heidelberg, Germany.
- Moshonas, M. G.; Lund, E.D.; Berry, R.E. and Veldhuis, M.K. (1972). Analysis of flavor constituents from lemon and lime. *J. Agric Food Chem.*, 20: 1029-1030.
- Moshonas, M., G. and Shaw, P. E. (1986). Quantities of volatile flavor components in aqueous orange essence and in fresh orange juice. *Food Technol.*, 40: 100-103.
- Moshonas, M. G. and Shaw, P.E. (1991). Ambersweet orange hybrid - compositional evidence for variety classification. *J. Agric. Food Chem.*, 39(8): 1416-1421.
- Moshonas, M. G. (1993). Classification of commercial orange juice products. *J. Agri. Food Chem.*, 41, 809.
- Moshonas, M. G. and Shaw, P. E. (1992). Comparison of static and dynamic headspace gas chromatography for quantitative determination of volatile orange juice constituents. *Lebensm. Wiss. u. Technol.*, 25, : 236 - 239.
- Moshonas, M. G. and Shaw, P. E. (1994). Quantitative determination of 46 volatile constituents in fresh, unpasteurized orange juice using dynamic headspace gas-chromatography. *J. Agric. Food Chem.*, 42, :1525-1528.
- Moukarzel, A. A. and Sabri, M.T. (1996). Gastric physiology and function: effects of fruit juices. *J. Am. Coll. Nutr.*, 15(5): 18s-25s.
- Nisperos-Carreido, M., O. and Shaw, P.E. (1990a) Volatile flavor components of fresh and processed orange juices. *Food Technol.*, 44 (4), 134-138.
- Nisperos-Carreido, M., O. and Shaw, P.E. (1990b). Comparison of volatile flavor components in fresh and processed orange juices. *J. Agric. Food Chem.*, 38, :1048 - 1052.
- Nisperos-Carreido, M., O.; Baldwin, E.A.; Moshonas, M., G. and Shaw, P. E (1992).

- Determination of volatile flavor components, sugars and ascorbic, dehydroascorbic and other acids in calamondin (*Citrus mitis*, Blanco). *J. Food Sci.*, 54, 227-230.
- Pala, M. and Bielig, H. J. (1978). Industriella Kon-centrierung und Aroma-Gewinnung von fluessigen Lebensmitteln. TU Berlin, Abteilung.Publikationen der Universitaetbibliotheek. C.F.: Askar, A.; El-Samahy, S.K.; Abd El-Baki, M.M; Ibrahim, S.S and Abd El-Fadeel, M.G. (1981). Production of lime juice concentrates using serum-pulp method. *Alimenta*, 20 , 121.
- Peleg, H. and Mannheim, L.H. (1970). Production of frozen orange juice concentrate from centrifugal separated serum and pulp. *J. Food Sci.*,35 : 649.
- Pfannhauser, W. (1989). Einsatz von Verbunds-Methoden Zur Qualitaets-beurteilung von Lebensmittel. Ein Ueberblick an Hand von Beispielen. *Ernaehrung*, 13, (4) :213-217.
- Pimazzoni, O. (1961). *Industria conserve* 36, 3 Cf . Askar, A.; El-Samahy, S.K.;Abd El-Baki, M.M; Ibrahim, S.S and Abd El-Fadeel, M.G. (1981). Production of lime juice concentrates using serum-pulp method. *Alimenta*, 20 , 121-12.
- Pino, J.; (1982). Correlation between sensory and gas-chromatographic measure ments on orange juices. *Acta Alimentaria*,11 (1), 1-9.
- Pino, J.; Toriclla, R. and Orsi, F. (1986). Correlation between sensory and gas-chromatographic measure ments on grapefruit juice volatiles. *Nahrung*, 30,:783-790.
- Rassis, D. and Saguy, I.S. (1995). Kinetics of aseptic concentrated orange juice quality changes during commercial processing and storage. *Int. J. Food Sci. Technology*, 30 (2): 191-198 .
- Rodrigues, P.A. and Culberton, C.R. (1983). Quntitative headspace analysis of selected compounds in equilibrium with orange juice.In Chharalambous, G., Inglett, G. (Eds) Academic Press, NewYork, vol. 2 pp. 187-195.
- Schiebrle, P. and Grosch, W. (1988). Identification of potent flavor compounds formed in an aqueous lemonoil/citric acid emulsion. *J. Agric. Food Chem.*, 36:797-800.
- Shaw, P. E. (1979). Quan-titative analysis of citrus essence oils. *J.*

- Agric. Food Chem., 27: 246-257.
- Shaw, P.E. (1987). Decreased hydrogen sulfide content in Valencia oranges after freeze damage. *Phytochemistry*, 23, 1175-1178.
- Shaw, P. E. (1991). Fruits II. In: Maarse, H. (Ed). *Volatile compounds in foods and beverages* Marcel Dekker Inc. p: 305-327
- Shaw, P.E, Busling, B.S. and Moshonas, M.G (1994). Classification of orange and grape fruit juices by pattern recognition Technique. *Fruit Processing*, 4 (2): 45-49.
- Sherif, E.A.A. (1999). Studies on some fruit juices. Ph.D. Thesis., Zagazig Univ. Faculty of Agric., Zagazig, Egypt.
- Velez, E. Costell; Orlando, L; Nadal; M.I.; Sendra; J.M and Izquierdo, L. (1993). Multidimensional scaling as a method to correlate sensory and instrumental data of orange juice. *J. Sci. Food Agric.*, 61, 41-46.

**Table 1:** Physicochemical properties of unpasteurized orange Juice and its pulp and serum.

Parameters	Unpasteurized orange juice	Pulp	Serum
Total soluble solids (°Brix)	11.20	-	10.00
Glucose (%)	2.45	1.36	2.25
Fructose (%)	2.40	1.99	2.20
Sucrose (%)	4.23	4.01	4.01
Titrateable acidity (%)	1.11	-	1.11
Free amino nitrogen (mg/100 ml)	32.25	-	-
Vitamin C (mg/ 100g)	55.61	13.23	42.38
Color	L	52.32	61.39
	a	8.99	17.35
	b	52.72	75.62
Cloud stability at 660 nm (%)	26.70	-	-
pH	3.33	-	3.31



**Table 2:** Concentration of volatile flavour compounds of unpasteurized Valencia orange juice, serum and pulp.

Flavour compounds (ppm)	Juice	Serum	Pulp
Acetaldehyde	6.3	5.7	5.6
Unkown 1	2.2	1.8	9.4
Methanol	Tr*	Tr*	Tr*
Ethylacetate	Tr*	Tr*	Tr*
Ethanol	411.0	522.0	422.0
$\alpha$ -Penine	0.4	Tr*	2.1
Iso-ethylbutanoate	Tr*	Tr*	Tr*
Unkown 2	0.1	0.1	Tr*
Ethylbutanoate	0.1	0.1	Tr*
Unkown 3	0.2	Tr*	1.0
Unknown 4	3.6	Tr*	22.4
Hexanal	Tr*	Tr*	Tr*
Limonene	119.0	Tr*	734.0
Unkown 5	0.5	Tr*	3.0
Ethylhexanoate	Tr*	Tr*	Tr*
Trans2-Hexnal	Tr*	Tr*	Tr*
1-Hexnanol	Tr*	Tr*	Tr*
Pelargon aldehyde	Tr*	Tr*	Tr*
Citronellal	Tr*	Tr*	0.4
Linalool	3.1	2.5	5.6
Valencene	0.4	Tr*	5.2
$\alpha$ -Terpineol	1.1	0.4	1.3
Citral /Geranial	Tr*	Tr*	Tr*
Citral / neral	Tr*	Tr*	Tr*

Tr\*: means concentration of a component is less than 0.1 ppm.

**Table 3:** Effect of enzymatic treatment on physicochemical properties of Valencia orange juice serum.

Parameters	Untreated	Enzyme concentration (ppm)		
		20	40	60
Total soluble solid (°Brix)	11.20	11.20	11.20	11.20
Titrateable acidity (%)	1.18	1.15	1.16	1.18
Brix / Acid ratio	9.50	9.70	9.70	9.50
pH	3.31	3.48	3.56	3.56
Viscosity (centipoise)	6.40	6.40	5.60	4.90
Total soluble solid (°Brix) after concentration	72.00	78.40	80.00	78.00

**Table 4:** Effect of enzymatic treatment of Valencia orange juice serum on volatile compounds of serum concentrates prepared by serum - pulp method .

Flavour compounds (ppm)	Untreated	Enzymatically treated
Acetaldehyde	16.0	4.8
Unkown 1	Tr*	Tr*
Methanol	Tr*	Tr*
Ethyacetate	Tr*	Tr*
Ethanol	15.0	2.7
$\alpha$ -Penine	Tr*	Tr*
Iso-ethylbutanoate	Tr*	Tr*
Unkown 2	0.9	1.0
Ethylbutanoate	Tr*	Tr*
Unkown 3	Tr*	Tr*
Unknown 4	Tr*	Tr*
Hexanal	Tr*	Tr*
Limonene	23.0	11.0
Unkown 5	Tr*	Tr*
Ethylhexanoate	Tr*	Tr*
Trans2-Hexnal	Tr*	Tr*
1-Hexnanol	Tr*	Tr*
Pelargon aldehyde	Tr*	Tr*
Citronellal	Tr*	Tr*
Linalool	Tr*	Tr*
Valencene	Tr*	2.0
$\alpha$ -Terpineol	Tr*	Tr*
Citral /Geranial	Tr*	Tr*
Citral / neral	Tr*	Tr*

Tr\*: means concentration of a component is less than 0.1 ppm.

**Table 5:** Effect of serum-pulp method on volatile flavour compounds of fresh unpasteurized and reconstituted orange juices.

Flavour compounds (ppm)	Fresh unpasteurized orange juice	Reconstituted juice from conventional method	Reconstituted juice from serum / pulp method	
			Untreated	Enzymatically treated
Acetaldehyde	6.3	0.7	1.3	0.9
Unknown 1	2.2	Tr*	0.8	Tr*
Methanol	Tr*	Tr*	Tr*	Tr*
Ethylacetate	Tr*	Tr*	Tr*	Tr*
Ethanol	411.0	Tr*	43.0	36.0
$\alpha$ -Penine	0.4	0.2	0.3	0.3
Trans2-hexenal	Tr*	Tr*	Tr*	Tr*
Unkown 2	0.1	0.9	Tr*	Tr*
Ethylbutanoate	0.1	Tr*	Tr*	Tr*
Unknown 3	0.2	Tr*	Tr*	Tr*
Unknown 4	3.6	1.7	3.1	2.9
Hexanal	Tr*	Tr*	Tr*	Tr*
Limonene	119.0	51.0	93.0	97.5
Unknown 5	0.5	0.2	0.4	0.3
Ethylhexanoate	Tr*	Tr*	Tr*	Tr*
Trans2-hexenal	Tr*	Tr*	Tr*	Tr*
Hexanol	Tr*	Tr*	Tr*	Tr*
Pelargon aldehyde	Tr*	Tr*	Tr*	Tr*
Citronellal	Tr*	0.1	Tr*	Tr*
Linalool	3.1	Tr*	0.8	1.1
Valencene	0.4	0.3	0.3	Tr*
$\alpha$ -Terpienol	1.1	Tr*	0.9 <sup>b</sup>	1.2
Citral /Geranial	Tr*	Tr*	Tr*	Tr*
Citral / Neral	Tr*	Tr*	Tr*	Tr*

Tr\* : means concentration of a component is less than 0.1 ppm.

## تأثير تركيز عصير برتقال الفالانشيا بطريقه السيرم - لب على خواص

### العصير ومركبات النكهة الطيارة

محمد عبد الحميد ربيع - صبحى سالم بسيونى

حسن ابراهيم صليحة - سامى محمد ابو المعاطى

قسم علوم الأغذية - كلية الزراعة - جامعة الزقازيق - الزقازيق

يتميز عصير البرتقال الطازج بنكهه جيده ولون جذاب وقيمة غذائية عالية ويقبل عليه المستهلك خاصة الذى يرغب فى المحافظة على وجبة صحية. وتعتبر مركبات عصير البرتقال من أهم منتجات البرتقال والذى له وضع مميز فى التجارة العالمية. يودى تركيز عصير البرتقال بالتبخير الى تغيرات فى خواص اللون والطعم والرائحة والقيمة الغذائية للعصير المسترجع. وقد تم فى هذا البحث دراسة استخدام المعاملة الإنزيمية بالاشتراك مع طريقه السيرم - لب لتركيز عصير البرتقال الفالانشيا وكذلك تأثير هذه العملية على التركيب الكيماوى ومركبات الرائحة الطيارة. وقد اختلفت الخواص الفيزيوكيماوية لكل من العصير واللب والسيرم لحد كبير فكانت النسبة بين الجلوكوز، الفركتوز والسكروز ٢:١:١ وكانت حبيبات اللب غنية فى محتواها من فيتامين ج وهو الاعلى من حيث قيم اللون كما أوضح التقدير الكمي لمركبات الرائحة الطيارة ان العدد الكلى لمركبات النكهة ٢٤ مركبا منها ٩ مركبات وجدت بتركيزات اعلى من ٠,١ جزء فى المليون وهى الاسيتالدهيد، الايثانول، الالفاتريبنول، اثيل بيوتانوت، الليمونين، لينالول، سيترونيلال، الفالانسين والالفابنين وعشره منها كانت موجوده بتركيزات اقل من ٠,١ جزء/مليون وهى الميثانول، الايثيل اسيتات، ايزوايثايل بيوتانوت، هكسنال، ايثايل هكسانوت، ترانس -٢- هكسنال، ١ - هكسانول، بلارجون الدهيد، جيرانيال ونيرال ولقد اوضح توزيع مركبات الرائحة الطيارة بين اللب والسيرم. ان الفابنين، الليمونين، الفالانسين، اللينالول و السيترونيلال كانت متواجدة فى حبيبات اللب بصفه اساسيه بينما الاسيتالدهيد، الايثانول و الايثايل بيوتانوت تواجدت أساسا فى السيرم. وبمقارنه مركبات الرائحة الطيارة للعصير غير المبستر والعصير المسترجع من مركز بطريقه التركيز العادية. والمسترجع من مركز بطريقه السيرم - لب وجد أن العصير الغير مبستر يتميز بأعلى محتوى من مركبات الرائحة وكان العصير المسترجع من طريقه السيرم - لب أعلى فى مكونات الرائحة من المسترجع من المركز بالطريقه العادية. وبناء على ذلك فان نتائج هذه الدراسة تعطى الدليل على ان طريقه السيرم - لب فعاله لإنتاج عصير نوجوده عالية.